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Asymmetric Formal [3+2] Cycloaddition Reaction of Isocyanoesters to 2-Oxobutenoate Esters by a Multifunctional Chiral Silver Catalyst

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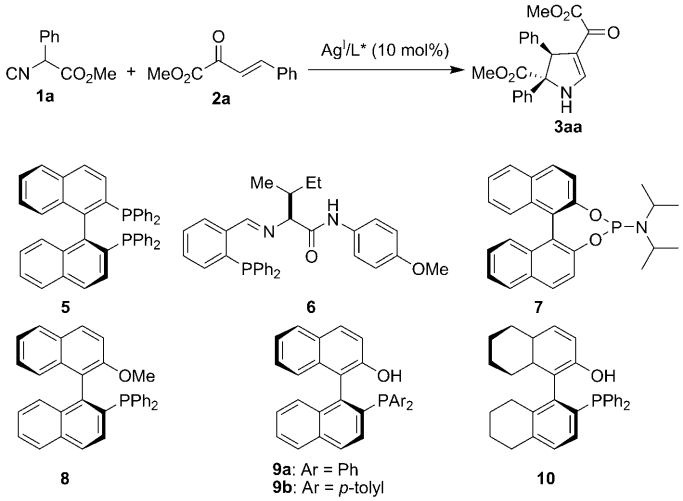
Chiral pyrrolidine constitutes a core structural motif prevalent in numerous natural alkaloids and pharmaceutically active substances and has proven widely applicable in total synthesis as a type of building block. In the last decade, great efforts have been devoted to the construction of optically pure pyrrolidine derivatives, leading to the appearance of many elegant catalytic methods.[1,2] Optically active dihydropyrroles possess a carbon–carbon double bond and thereby can be adapted for diverse synthetic purposes by exploiting a wide range of reactions that are compatible with olefins.[3] However, approaches to access the molecules of this type in an enantioselective fashion are rather limited, but include the asymmetric Heck reaction of dihydropyrroles with halogenated (or pseudo-halogenated) compounds,[4] partial hydrogenation of trisubstituted pyrroles,[5] and a sequential process of *N*-butoxycarbonyl (Boc)-protected imines with propargylated malononitriles (recently discovered by Jørgensen).[6] Specifically, formal cycloaddition reactions of metalated isocyanides to electronically poor olefins offers the most straightforward method to produce highly functionalized 2,3-dihydropyrroles in comparison with other alternatives.[7] However, the enantioselective variants have not been available until our recent report of a highly enantioselective organocatalytic formal cycloaddition reaction of α-substituted isocyanoesters with nitroolefins by cinchona alkaloid derivatives.[8] Unfortunately, the expansion of this protocol to an unsaturated carbonyl compound **2a** provided a moderate yield with low levels of stereoselectivity.[9] To address this challenge, and inspired by the asymmetric ferrocenyl–gold(I)/silver(I)-catalyzed aldol reactions of isocyanoacetate and tosylmethyl isocyanides with aldehydes,[10,11] we turned our attention to a Lewis-acid-catalyzed asymmetric version of the cyclization between isocyanoesters and 2-oxobutenoate esters. Although a similar reaction catalyzed by silver acetate has been reported,[12] no enantioselective version is available. Herein, we report the first highly enantioselective formal [3+2] cycloaddition reaction of isocyanoesters to 2-oxobutenoate esters catalyzed by a chiral silver complex, which performed as a multifunctional catalyst to control the stereochemistry.

Our initial investigation focused on screening chiral ligands (Scheme 1) for the silver-acetate-catalyzed cycloaddition reaction of methyl α-phenylisocyano acetate (**1a**) with methyl 2-oxo-4-phenylbutenoate (**2a**) conducted in dichloromethane at 0 °C.[13] Although high yields were obtained, disappointingly all the chiral ligands, including (*S*)-BINAP **5**,[14] Hoveyda ligand **6**,[15] (*R*)-phosphoramidite ligand **7**,[16] and (*S*)-MOP **8**–**10**,[17] offered racemic products. Inspired by the work of Hayashi and co-workers,[11a] which indicated that the selective generation of a tri-coordinated silver(I) catalyst would bring about higher stereoselectivity through slow addition of isocyanoacetate to a solution of the silver(I) catalyst and substrates, we then examined our ligands again through slow addition of isocyanoacetate over two hours (see the Supporting Information). To our great delight, Hoveyda ligand **6** (the analogues of which have provided high stereoselectivity with Ag^I and Cu^I for other type of reactions)[15] gave an improved result (26% enantiometric excess (*ee*) for the major diastereomer, Table 1, entry 2). No significant enhancement in the *ee* value was achieved by using other ligands except those with a hydroxyl group, as shown



Scheme 1. Chiral ligands evaluated for the titled reaction.

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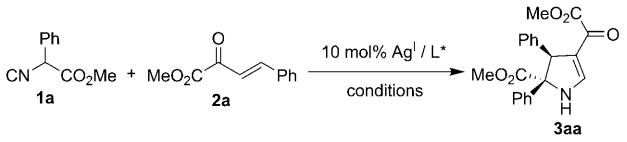
Table 1. Evaluation of ligands (L*) and optimization of reaction conditions.[a]



Entry	Ag^I	L*	Solvent	Yield [%][b]	d.r.[c]	*ee* [%][d]
1	AgOAc	**5**	CH_2Cl_2	91	2:1	6 (0)
2	AgOAc	**6**	CH_2Cl_2	80	2:1	26 (10)
3	AgOAc	**7**	CH_2Cl_2	96	1:1	3 (2)
4	AgOAc	**8**	CH_2Cl_2	94	2:1	5 (0)
5	AgOAc	**9a**	CH_2Cl_2	95	2:1	82 (72)
6	CF_3CO_2Ag	**9a**	CH_2Cl_2	82	1:1	27 (41)
7	Ag_2CO_3	**9a**	CH_2Cl_2	87	1:1	42 (51)
8	AgOTf	**9a**	CH_2Cl_2	n.r.[f]	–	–
9	$PhCO_2Ag$	**9a**	CH_2Cl_2	88	3:1	87 (84)
10[e]	AgOAc	**9a**	CH_2Cl_2	95	2:1	90 (70)
11[e]	AgOAc	**9b**	CH_2Cl_2	86	3:1	88 (80)
12[e]	AgOAc	**10**	CH_2Cl_2	90	3:1	88 (81)
13[e]	AgOAc	**9a**	$(ClCH_2)_2$	95	3:1	88 (78)
14[e]	AgOAc	**9a**	$CHCl_3$	90	3:1	92 (83)
15[e]	AgOAc	**9a**	CCl_4	55	4:1	84 (56)
16[e]	AgOAc	**9a**	toluene	99	3:1	80 (56)
17[e]	AgOAc	**9a**	THF	90	3:1	84 (68)
18[e]	AgOAc	**9a**	CH_3CN	95	1:1	46 (39)

[a] Reaction conditions: **1a** (0.1 mmol), **2a** (0.1 mmol), AgOAc (10 mol%), and ligand (12 mol%). Isocyanoacetate **1a** was added by a syringe pump over a period of 2 h to a solution of **2a** and catalyst at 0°C. [b] Yields of isolated products. [c] Determined by ^{1}H NMR spectroscopy. [d] Determined by HPLC analysis; *ee* values shown in parentheses for the minor diastereomer. [e] at −10°C. [f] n.r. = no reaction.

in **9** and **10**. Most interestingly, the chiral ligand **9a** offered remarkably higher levels of enantioselectivity than **8**, clearly demonstrating that the presence of a hydroxyl group is crucial to the induction of high enantioselectivity (Table 1, entries 4 and 5). In addition, these results suggest that the hydrogen-bonding interaction between the hydroxyl of the ligand **9a** and the substrate is important to control the stereoselectivity. The similar hydrogen-bonding interaction between **9a** and substrates was also found in organocatalyzed Morita–Baylis–Hillman reactions.[18] Fortunately, a crystal of the complex formed from silver acetate and (*S*)-**9a** was obtained and its X-ray structure acquired (Figure 1). To the best of our knowledge, this is the first silver crystal of such type with an additional hydroxyl Brønsted acid functionality.[19] In this complex, only the phosphorus coordinates to the silver, whereas the hydroxyl proton forms a hydrogen bond with one of the acetate oxygens. As such, the silver complex might work as a multifunctional catalyst in the cyclization reaction by acting as a silver(I) Lewis acid, an acetate base, and proton as a Brønsted acid capable of activating substrates by a hydrogen-bonding interaction.[18,20] Variation of the silver source revealed that the basicity of the counter ion in the silver catalyst precursors not only plays an important role in the catalytic activity, but also is crucial to the enantioselectivity (Table 1, entries 5–9). Thus, AgOAc, AgTFA, Ag_2CO_3, and $PhCO_2Ag$ all exhibited excellent catalytic activity in combination with **9a**. In contrast, AgOTf, which proved to be an excellent silver catalyst precursor in asymmetric aldol reaction of tosylmethyl isocyanide with aldehydes,[11b] was unable to activate the nucleophile **1a** and hence was catalytically inactive for the reaction, presumably due to the much lower basicity of the triflate than other counter ions. In terms of the stereoselectivity, AgOAc emerged as the catalyst precursor of choice. The ligand **9a** could offer 90% *ee* upon conducting the reaction at −10°C, whereas its structural analogues **9b** and **10** showed slightly lower enantioselectivities (Table 1, entries 10–12). A survey of solvents identified chloroform as the most suitable media; the highest level of stereoselectivity was observed in these conditions (92% *ee* for the major diastereomer, Table 1, entries 13–18). The absolute configuration of **3aa** was assigned by X-ray crystal structure analysis (see the Supporting Information).

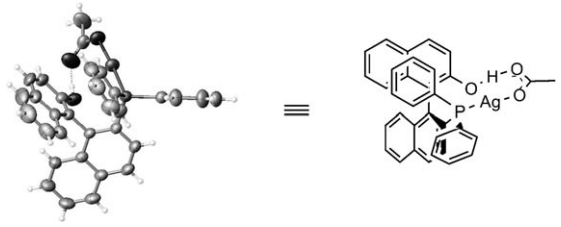
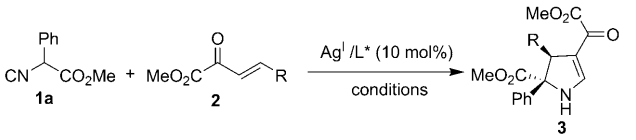
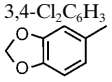


Figure 1. Crystal structure of the silver acetate complex with (*S*)-**9a**.

Having established the optimal conditions, the generality of this reaction between α-phenylisocyano acetate (**1a**) and 2-oxobutenoate esters was next explored (Table 2). Either electronically rich or electronically poor phenyl-substituted substrates underwent smooth cyclization reactions to give

Table 2. Scope for 2-oxobutenoate esters.[a]

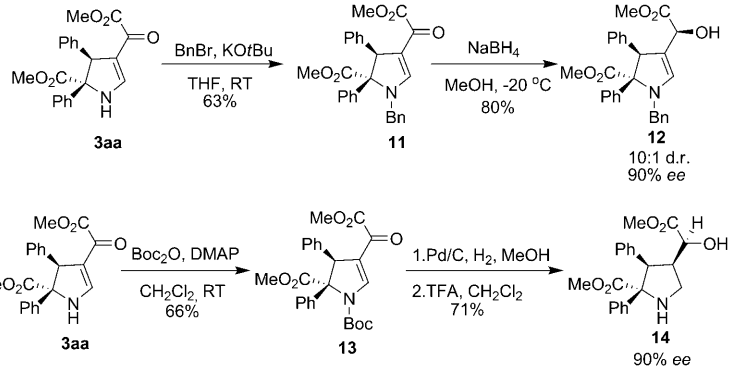


Entry	**3**	R	Yield [%][b]	d.r.[c]	*ee* [%][d]
1	**3ab**	4-ClC_6H_4	91	3:1	94 (75)
2	**3ac**	4-BrC_6H_4	81	4:1	90 (74)
3	**3ad**	4-FC_6H_4	73	3:1	92 (90)
4	**3ae**	4-MeC_6H_4	95	3:1	91 (84)
5	**3af**	4-CNC_6H_4	84	4:1	91 (71)
6	**3ag**	3-ClC_6H_4	91	3:1	90 (75)
7	**3ah**	3-$MeOC_6H_4$	85	3:1	94 (78)
8	**3ai**	3,4-$Cl_2C_6H_3$	98	4:1	93 (81)
9	**3aj**		94	3:1	91 (83)
10	**3ak**	2-furyl	90	2:1	93 (91)
11	**3al**	2-naphthyl	96	2:1	90 (78)
12	**3am**	*c*-C_6H_{11}	86	5:1	93 (80)

[a] Reaction conditions: **1a** (0.1 mmol), **2a** (0.1 mmol), AgOAc (10 mol%), and ligand (12 mol%) in $CHCl_3$ at −10°C. Isocyanoacetate **1a** was added by a syringe pump over a period of 2 h to a solution of **2a** and catalyst. [b] Yields of isolated products. [c] Determined by ^{1}H NMR spectroscopy. [d] Determined by HPLC analysis; *ee* values are shown in parentheses for the minor diastereomer.

the 4,5-dihydropyrroles, whereas both reaction conversion and stereoselectivity showed dependence to some degree on the electronic feature of substituents on the phenyl substituent. The 2-oxobutenoate, with a relatively electronically rich *para*-methylphenyl substituent, provided a comparably good yield and stereochemical outcome like those with electronically deficient groups (Table 2, entry 4, entries 1–3, and 5). *Meta*-substituted phenyl 2-oxobutenoates underwent the cyclization reaction with high levels of enantioselectivity (Table 2, entries 6 and 7). Disubstituted phenyl 2-oxobutenoates participated in the reaction to provide excellent yields and high levels of enantioselectivity for the major diastereomers (Table 2, entries 8 and 9). A heteroaryl 2-oxo-butenoate is also a good substrate in a high yield and excellent enantioselectivity (Table 2, entry 10). The reaction occurred nicely using 2-oxobutenoate with a 2-naphthyl substituent and also gave high enantioselectivity, albeit with a comparably lower diastereomeric ratio (d.r., Table 2, entry 11). The cyclohexyl 2-oxobutenoate reacted well with **1a** to give a high yield and with a slightly higher d.r. of 5:1 and 93% *ee* for the major diastereomer (Table 2, entry 12).

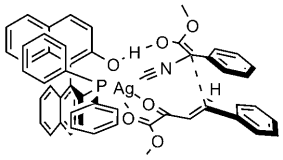
A further study on the substrate scope was then focused on the tolerance of the α-substituted isocyanoacetates (Table 3). A variety of α-arylisocyano acetates could be tolerated in the reaction. Either electronically rich or electronically poor α-arylisocyano acetates participated in the cyclization reaction in high yields and with excellent levels of enantioselectivity. Particularly, both the 4-chloro- and 4-acetoxyphenyl isocyanoacetates afforded 98% *ee* (Table 3, entries 2 and 4).



Scheme 2. Stereoselective reductions of the dihydropyrrole into functionalized hydroxyacid esters. Bn = benzyl; Boc = *tert*-butoxycarbonyl; DMAP = 4-dimethylaminopyridine.

The 2,3-dihydropyrrole contains multiple functionalities and thereby can be readily transformed into structurally diverse pyrrolidine derivatives through well-established methods. For example, 2,3-dihydropyrrole **3aa** was first protected with a benzyl (Bn) group by exposure to benzylbromide in the presence of potassium *tert*-butoxide (Scheme 2). Then, treatment with sodium borohydride followed to yield dihydropyrrole **12** in high yield with a high diastereomeric ratio. The hydrogenation of the Boc-protected product **13** was able to generate pyrrolidine **14** with maintained *ee* values under the catalysis of Pd/C. Both dihydropyrroles of type **12** and pyrrolidines of **14** (Scheme 2), which contain useful hydroxy ester functional groups, show potential value in the design of new catalysts[21] and occur frequently in many biologically active molecules.[1,22]

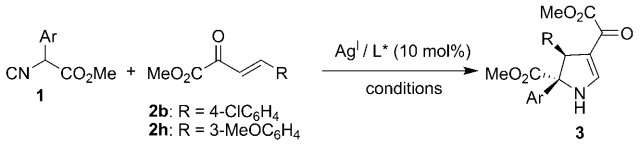
According to the crystal structure of the silver(I)/(*S*)-**9a** complex (Figure 1) and previous reports on related transformations,[10,11a,23] we proposed a transition structure to explain the observed stereochemistry (Scheme 3). α-Deprotonation



Scheme 3. Proposed transition state to explain the stereochemistry observed.

of metallated isocyanide with the acetate anion generates an enolate-like species, which is stabilized by a hydrogen bond formed with the OH group on the catalyst. Simultaneously, both carbonyl groups of the 2-oxobutenoate ester are able to coordinate with the Ag center[24] and thus lower the LUMO of the carbon–carbon double bond, which becomes more electrophilic toward the enolate-like nucleophile from its *Re* face, to facilitate an enantioselective Michael addition and give the pyrrolidine products with high levels of stereochemical control.

Table 3. Scope of α-substituted isocyanoacetates.[a]



Entry	3	Ar	Yield [%][b]	d.r.[c]	*ee* [%][d]
1	**3bh**	3-ClC_6H_4	78	3:1	93 (84)
2	**3ch**	4-ClC_6H_4	93	4:1	98 (70)
3	**3dh**	4-$MeOC_6H_4$	78	3:1	96 (82)
4	**3eh**	4-$AcOC_6H_4$	81	3:1	98 (86)
5	**3fb**	4-FC_6H_4	86	3:1	93 (76)

[a] Reaction conditions: **1a** (0.1 mmol), **2a** (0.1 mmol), AgOAc (10 mol%), and ligand (12 mol%) in $CHCl_3$ at −10 °C. Isocyanoacetate **1a** was added by a syringe pump over a period of 2 h to a solution of **2a** and catalyst. [b] Yields of isolated products. [c] Determined by ^1H NMR spectroscopy. [d] Determined by HPLC analysis; *ee* values are shown in parentheses for the minor diastereomer.

In conclusion, we have established the first chiral Lewis-acid-catalyzed formal [3+2] cyclization reaction of isocyanoesters with 2-oxobutenoate esters in high yields and excellent levels of enantioselectivity by using a silver complex of silver acetate and (*S*)-(2′-hydroxy-1,1′-binaphthyl-2-yl) diphenylphosphine. Most specifically, this is the first successful application of a silver-OH-MOP complex in asymmetric catalysis. In addition, we have also provided evidence that the functionality of hydroxyl in this complex plays a crucial role in the control of stereoselectivity. The crystal structure of the complex suggested that it works as a multifunctional catalyst in the asymmetric cyclization reaction. These findings may reveal a strategy for designing new catalysts for this class of cyclization reactions.

Experimental Section

General procedure: Under Ar, a solution of AgOAc (0.01 mmol) and chiral ligand **9a** (0.012 mmol) in $CHCl_3$ (0.5 mL) were stirred at room temperature for 1 h. Then, a solution of **2** (0.1 mmol) in $CHCl_3$ (1.0 mL) was added. After cooling to −10 °C, a solution of **1** (0.1 mmol) in $CHCl_3$ (1.0 mL) was added by a syringe pump over a period of 2 h to a solution of **2** and the catalyst. After the completion of reaction (monitored by TLC), the mixture was purified by column chromatography to give the product.

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Keywords: asymmetric catalysis · cycloaddition · isocyanoesters · multifunctional · silver

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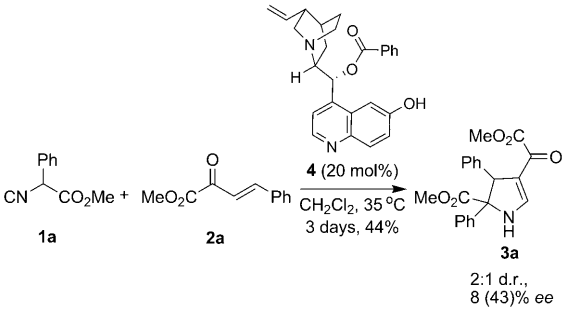

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